

plate, it is very definite.) The acetic acid in 5 g. of sample is estimated on preceding page.

A sample of carbonate of lime (about 40.00 per cent CO_2), part of the residue from the manufacture of acetone by heating calcium acetate, was treated by the above method and the following figures obtained:

N/10 $\text{Ba}(\text{OH})_2$ used.....	97.09 cc.
N/10 HCl to titrate back.....	67.02 cc.
N/10 $(\text{BaOH})_2$ equiv. to CO_2 plus acetic acid	30.07 cc.
N HCl added.....	5.63 cc.
N NaOH to titrate back.....	2.63 cc.
N HCl equiv. to CO_2	3.00 cc. or 30 cc. N/10 HCl
30.07 — 30.00 cc. = 0.07 cc. N/10 $(\text{BaOH})_2$ or 0.008 per cent acetic acid	

As this sample had been re-heated for a long time before this analysis was made, the chance of any acetate still being present was reduced to a minimum. The presence of a large amount of CO_2 therefore does not seem to interfere with the acetic acid determination.

While the above procedure appears somewhat complicated at first glance, it does not prove to be so, as the distillation after once it is adjusted, needs very little attention, thus allowing the person in charge to attend to other duties.

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THE DETERMINATION OF ACETONE

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The purpose of this investigation was to find an accurate method for the determination of acetone in methyl alcohol. All of the published methods were investigated with results as follows:

MESSINGER'S METHOD¹ is a volumetric method depending upon the reaction between iodine and acetone to form iodoform. Messinger claims that accurate results can be obtained by this method. A criticism by Vaubel and Scheuer² of this method is that when thiosulfate is used in titrating back, the results are always too low, and, therefore, they recommend the use of arsenious acid instead of the thiosulfate. The writer tested this method, using thiosulfate as well as the arsenious acid, but could not get accurate results. The precautions recommended by Collischon³ were observed but the results were not satisfactory. I found that if, after the addition of the N/5 iodine solution, the mixture is shaken for 5 min., a low result is obtained, while shaking for 20 min. gives a higher result, the percentage varying with the length of time of agitation. These experiments were tried on a pure acetone solution without the presence of alcohol. When methyl alcohol was present the percentage of acetone found was several per cent higher. The increase could not be due to the alcohol furnishing the extra amount of acetone as the quantity it contained was duly considered.

¹ Ber., 21 (1888), 3366.

² Z. angew. Chem., 18 (1905), 214.

³ Z. anal. Chem., 29 (1890), 562.

THE ROBINEAU AND ROLLINS METHOD as modified by L. F. Kebler¹ was found by the author to give the most accurate results. The presence of alcohol and the time of shaking did not influence the percentage of acetone.

The acetone that was used in trying out these methods was Merck's reagent guaranteed by them to be 99 to 100 per cent. The percentages of acetone obtained in four determinations using the Robineau and Rollins method were as follows:

Determination No.	I	II	III	IV
Acetone, per cent.	96.37	96.37	95.89	95.74

There remained 4 per cent unaccounted for. The Bureau of Standards at Washington advised checking the purity of the acetone by means of the specific gravity and referred to Timmerman² as giving the best value obtainable.

The specific gravity I found was 0.80716 $15^\circ/4^\circ$ and Timmerman's value is 0.79574 $15^\circ/4^\circ$, which would indicate the presence of water. Krug and McElroy³ found that at 20°C. , one per cent of water raises the specific gravity 0.0031. Squibb⁴ found that at 15° one per cent of water raised the specific gravity 0.0029. The specific gravity of Merck's acetone was 0.80716 $15^\circ/4^\circ$, therefore, there could be 3.68 per cent of water present, assuming as correct Timmerman's value for anhydrous acetone.

The author wishes to express his thanks to Dr. I. W. Fay, of the Polytechnic Institute of Brooklyn, for his advice during the work.

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SOME RESULTS OF ANALYSIS OF AIRS FROM A MINE FIRE

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There is no doubt that gas analysis has found considerable use in locating mine fires and especially in following the progress of these underground fires. Not a great deal of data has been published, however. The writers, therefore, considered it proper that they publish some data which may be of interest to those connected with mining work, particularly with anthracite coal mining.

The mine at which the samples were taken is an anthracite coal mine, a mine considered as a rather gaseous mine, or one generating a fairly large quantity of methane or fire damp.

On November 18, 1916, several men were overcome while at work inside the mine. On November 19, a fire was discovered. Batteries were erected for the purpose of smothering out the fire by cutting off all the fresh air supply.

In order to carry out this investigation pipes were extended through the batteries, these pipes being closed by means of valves at the outer ends. From time to

¹ J. Am. Chem. Soc., 19 (1897), 316.

² Bul. soc. chim. belg., 24 (1910), 263.

³ J. Anal. Appl. Chem., 6 (1892), 187.

⁴ J. Am. Chem. Soc., 17 (1895), 200.